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INVESTIGATION OF TIN AS A CONSTITUENT OF INORGANIC COATINGS FOR MAGNESIUM ALLOYS

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SermeTel, Incorporated

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INVESTIGATION OF TIN AS A CONSTITUENT OF INORGANIC COATINGS FOR MAGNESIUM ALLOYS

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FINAL REPORT

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bу

Mark F. Mosser

Submitted to the

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for

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coatings; these showed little corrosion resistance. Modification of alkali silicate/tin coatings were made with soluble salts to cause controlled leaching; these salts included amine salts, permanganates, and tin compounds. Results indicated enhanced corrosion. Strontium chromate and EDTA salts were used in primer coatings; these coatings showed some improvement in salt spray resistance. Additions of alkaline leaching materials were made consisting of magnesium oxide and aluminum hydroxide in the alkali silicate/tin coatings. These showed slightly improved salt spray resistance. When tested as primer coating with paint systems failure resulted during heat cycle/salt spray testing. No suitable coating based on tin was produced by the investigation.

FOREWARD

This Report was prepared by SermeTel Incorporated under U.S. Naval Air Systems Command Contract N62269-74-C-0548. This contract was initiated and administered by the Naval Air Development Center, Warminster. The work performed under this contract covers the period 1 May 1974 -30 April 1975.

Written by:

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SUMMARY

A series of tests was initiated to evaluate tin as a filler material in inorganic coatings to be used as a coating for magnesium alloys. Coatings were made based on tin powder incorporated in alkali silicate, quaternary ammonium silicate and chromate/phosphate binders. Tests were run evaluating zinc immersion, stannate, pyrophosphate, and galvanic anodize pretreatments. Salt spray testing indicated alkali silicate coatings with galvanic anodize pretreatment showed improved results. Tin-cadmium and tin-zinc alloys were tested in place of tin powder. Tests were run on ethyl silicate based coatings; these showed little corrosion resistance. Modification of alkali silicate/til coatings were made with soluble salts to cause controlled leaching; these salts included amine salts, permanganates, and tin compounds. Results indicated enhanced corrosion. Strontium chromate and EDTA salts were used in primer coatings; these coatings showed some improvement in salt spray resistance. Additions of alkaline leaching materials vere made consisting of magnesium oxide and aluminum hy 'roxide in the alkali silicate/tin coatings. These showed slightly improved salt spray resistance. When tested as primer coating with paint systems failure resulted during heat cycle/salt spray testing. No suitable coating based on tin was produced by the investigation.

PREFACE

Protective coatings for metallic substrates operate based on one or more basic principles. First, a coating can be of the polymeric pore-free type operating as a barrier. Secondly, a coating can contain a more galvanically active metal and protect the substrate by preferential attack by the corrosive medium on the more active metal in the coating. Third, a coating may contain a slightly soluble salt which leaches out of the coating, modifying the corrosive conditions and passivating the substrate.

The inorganic binders commonly used for coating on metals are normally silicates, silica gel, or chromate/phosphate based. These all are inherently porous. Operation by the barrier principle is, as a result, unlikely. Since in this investigation the metallic substrate to be protected is magnesium alloy, use of a sacrificial type coating approach is extremely remote. As a result, the approach must be made based on leaching a salt from the coating that produces conditions not conducive to corrosion.

Tin as a filler is relatively compatable with magnesium in a galvanic series. Other metals of similar galvanic position are cadmium, zinc and aluminum. This investigation is based on this relative galvanic compatability of tin and magnesium and the use of a leaching effect in protecting magnesium.

INTRODUCTION:

The preliminary requirements for a tin based coating for magnesium alloys involved a coating that preferably could be air dried and resist prolonged exposure to 400°F. In addition, a high order of salt spray resistance, good adhesion, reasonable smoothness, and abrasion resistance were desired.

All coatings made were applied to AZ31B alloy using standard spraying equipment. All reagents used were of A.C.S. grade where applicable and of highest available purity in those cases where no A.C.S. grades were available. Tin powder used was 99.9% tin and nominally 1-5 microns in size. Tin alloy certifications are given in Appendices A and B.

PRELIMINARY INVESTIGATION

Binder Evaluation

A series of six (6) binders was selected for preliminary study as possibly applicable to the problem:

1. SermeTel 421 - Alkali silicate based binder, pH 11.0

- 2. Sodium Silicate/Quaternary ammonium silicate based binder pH 10.5 (Quram 3365, Philadelphia Quartz Co.)
- 3. Quaternary ammonium silicate binder pH 10.8 (Quram 220, Philadelphia Quartz Co.)
- 4. SermeTel Binder Chromate/Phosphate based binder pH 1.6
- 5. SermeTel Binder Chromate/Phosphate based binder pH 2.4
- SermeTel 469 Chromate modified alkali silicate binder pH 10.9

As an initial test, grit blasted AZ31B-H24 panels were immersed in each of the listed binders and observed for 2 hours. Results of the test show all binders to have some reactivity with the alloy.

- 1. SermeTel 421 medium brown discoloration of the panel
- 2. Sodium Silicate/Quaternary Silicate Slow formation of black intergranular corrosion.
- Quaternary Silicate Slow formation of black intergranular corrosion.
- 4. Chromate/Phosphate (pH 1.6) rapid attack
- 5. Chromate/Phosphate (pH 2.4) rapid attack
- 6. SermeTel 469 rapid formation of black intergranular corrosion.

From these results binder #1 showed least tendency to react with magnesium alloy. Since binders #1, 2, 3, and 6 dry within fifteen minutes, these all could be used as binders.

One additional immersion test was run incorporating 0.5% by weight potassium permanganate (KMnO4) into binder 1. Two hour immersion produced no effect on the panel.

Binder/Tin Evaluation

Each of the six (6) binders being evaluated were mixed with tin powder (1-5 microns 99.9%) in the ratios of 1 gram per milliliter and 2 grams per milliliter. No reactions were observed in any of the binders. Long term standing produced gelation of binders 2 and 3 which have a six month shelf-life when stored in original containers; the others were usable 11 months after mixing.

Panel Pretreatment

Because of the reactivity of the binders tested with the AZ31B substrate, a series of tests were initiated to find a suitable compatable pretreatment that would aid in passivating the surface and provide a coating base.

Zinc Immersion Coating

Solutions for z' immersion coating were prepared using the data of refere $-\frac{1}{2}$ 1.

Activating Pickle

757 ml phosphoric acid (85%) 400 grams sodium bifluoride water to one (1) gallon

Zinc Immersion Coating Bath
120 grams zinc sulfate

480 grams sodium pyrophosphate

20 grams sodium fluoride

20 grams potassium carbonate

water to one (1) gallon

Panels of AZ31B alloy were grit blasted then immersed in the activating pickle for 1 minute at ambient temperature. They were then rinsed with water, dried, followed by immediate immersion in the zinc immersion coating bath. The bath was agitated by stirring and heated to 180°F. Immersion times were 3-6 minutes. Coatings produced were gray and adherent.

Stannate Coatings

A solution was prepared utilizing the stannate bath noted in reference #1.

Stannate Bath

16 grams sodium hydroxide

80 grams potassium stannate, dihydrate

80 grams sodium pyrophosphate

1428 ml water

Panels prepared by grit blasting were immersed for one (1) minute in the same activating pickle used for the zinc immersion coating. The panels were then rinsed, dried, and immersed for two (2) minutes at 180°F in the stannate bath. A dark gray film formed on the alloy.

Because of the similarity of stannate coatings and the coatings being investigated, another example was prepared using stannous pyrophosphate2:

50 grams sodium pyrophosphate

50 grams stannous pyrophosphate

10 grams dextrine

water to one (1) liter

⁽¹⁾ White, E.L. and F.W. Fink Corrosion protection of Magnesium and Magnesium alloys - DMIC Memorandum 205 Defense Metals Information Center Battelle Memorial Institute, Columbus, Ohio pg A-4, D-1.

⁽²⁾ Doss, Jodie, Multimetal Coating Process for Composite Metal Assemblies. Rock Island Arsenal Technical Report 65-1137 Rock Island, Illinois pg 14.

This bath was adjusted in pH to 8.0 with sodium hydroxide. Panels activated by immersion in the activating pickle previously described were rinsed and immersed for 2 minutes at 195°F in the pyrophosphate bath. Resulting coatings were black and adherent. Galvanic Anodize

A third type of pretreatment prepared was galvanic anodize MIL-M-3171 type IV. The solution was made up as follows:

120 grams ammonium sulfate 120 grams sodium dichromate 15 mls ammonium hydroxide water to one (1) gallon

Grit blasted AZ31B panels were immersed for 20 minutes at 135°F, coupling the panels to a stainless steel beaker holding the bath. Panels were then rinsed three times in running water and dried. Resulting coatings were black and adherent. Grit Blasted

A fourth preparation consisted only of grit blasting after degreasing. Such a prepared surface by reason of its high activity was used as a point of comparison to determine the extent of deactivation produced by the previously listed pretreatment procedures.

Evaluation of Coatings and Pretreatments

Panels of AZ31B-H24 were cut into 2" x 4" specimens, deburred, degreased, grit blasted, then treated with the pretreatments listed. Following drying all were exposed to 5% salt fog per ASTM B117-73. Panels were tested as treated and with scribed "X" on the panels. Salt fog exposure was for 24 hours. Results indicated the following sequence of corrosion resistance:

- a. galvanic anodize (best)
- b. stannous pyrophosphate
- c. stannata
- d. zinc immersion
- e. bare (worst)

Using the six binders described, coatings were prepared using 2 grams tin powder (1-5 microns) per milliliter of binder. These six coatings were applied to AZ31B-H24 panels that had been treated with the four (4) pretreatments listed. Application was by standard spraying techniques. Panels 1-3 and 6 were cured at 175°F for 2 hours, panels 4 and 5 had an additional cure at 650°F for 1 hour.

flexibility - panels were bent over a mandrel having a diameter 20X the thickness of the panel. Panels were then examined for loss of adhesion on the convex side of the panel only.

thickness - thicknesses of coatings were determined using a Biddle Eddy current Gauge (Model 285)

appearance - panels were examined for evidence of reaction of the coating with the substrate using visual observation with a low power (30X) binocular microscope.

salt fog testing - one panel of each type was placed in a salt fog chamber and exposed for 24 hours in accordance with ASTM B117-73.

Results are listed in Tables 1 and 2

TAL. I Results of Preliminary Testing One (1) coating thickness on AZ31B

ė,					
Appearance All are uniformly gray in color with no signs of reaction with the substrate	All are uniformly gray except the bare panel which shows signs of reaction	All are uniformly gray except the bare panel which shows slight reactivity	Signs of reaction with the substrate on all panels; the bare panel shows most reaction	Some reaction with the substrate on all panels; the bare panel showed considerable reaction	All are uniformly gray except the bare panel which shows discoloration in spots.
Flexibility pass pass pass pass pass	pass pass pass fail	pass pass pass pass	pass pass pass pass	pass pass pass fail	pass pass pass pass
Mils Thickness 2.4 3.1 2.8 2.4 1.8	6.6.6.9.9.9.9.9.9.9.9.9.9.9.9.9.9.9.9.9	2.02.1.8	3.5.4.6.	2.7 2.5 2.3	2.6 2.7 2.6 3.6
Pretreatment Zinc immersion Stannate Pyrophosphate Galvanic Anodize Bare	Zinc immersion Stannate Pyrophosphate Galvanic Anodize Bare	Zinc immersion Stannate Pyrophosphate Galvanic Anodize Bare	Zinc immersion Stannate Pyrophosphate Galvanic Anodize Bare	Zinc immersion Stannate Pyrophosphate Galvanic Anodize Bare	Zinc immersion Stannate Pyrophosphate Galvanic Anodize Bare
Binder 1. SermeTel 421	2. Sodium/Quaternary Silicate	3. Quaternary Silicate	4. Chromate/Phosphate pH 1.6	5. Chromate/Phosphate pH 2.4	6. SermeTel 469

TABLE II
Results of Preliminary Testing
Salt Fog Test Results at 24 hours

Binder # 1 2 3 4 5 6	Pretreatment Zinc immersion Zinc immersion Zinc immersion Zinc immersion Zinc immersion Zinc immersion	Results C B D F C
1 2 3 4 5 6	Stannate Stannate Stannate Stannate Stannate Stannate	C D E E D
1 2 3 4 5 6	Pyrophosphate Pyrophosphate Pyrophosphate Pyrophosphate Pyrophosphate Pyrophosphate	B C D E F D
1 2 3 4 5 6	Galvanic anodize Galvanic anodize Galvanic anodize Galvanic anodize Galvanic anodize Galvanic anodize	A C F F C
1 2 3 4 5 6	Bare Bare Bare Bare Bare Bare	6 6 6 6 6

Letter designations A through G are used to tabulate results. The letter "A" represents the best panel while G represents the worst. In this test "A" represents slight corrosion with the coating intact. G represents catastrophic failure of coating and panel.

Results of Preliminary Investigation

Based on the tests run the following conclusions can be made:

- While none of the binders tested react with tin, several react with the magnesium alloy substrate even if there is a suitable pretreatment.
- Most show good adhesion and flexibility but salt fog testing results are more dependent on pretreatment than which coating was applied.
- 3. Binder types 4 and 5 can be eliminated due to the high cure temperature required and the tendency to react with the alloy substrate even through neutral or alkaline pretreatments.
- 4. Of pretreatments used only the galvanic anodize pretreatment showed any salt fog resistance indicating little if any useful activity of the tin filler.

BINDER/FILLER MODIFICATION

Tin Alloy Fillers

Two alloys were chosen for study based on galvanic compatability with both tin and magnesium.³ These alloys are the tin-cadmium eutectic alloy (33% Cd) and the tin-zinc eutectic alloy (8% Zn).⁴ These were commercially prepared and furnished - 200 mesh. Analyses are given in Appendices A and B.

The tin-cadmium alloy was tested by incorporating it in binder #1 - SermeTel 421 in the ratio of 2 grams per milliliter. No reaction was noted. This coating was spray applied to AZ31B panels, some grit blasted and others galvanic anodize pretreated. Panels of each group were cured at 160°F overnight, other panels were cured at 350°F for two (2) hours, other panels at 400°F for 2 hours.

Examples of each were examined for flexibility, reaction of the coating with the substrate, and salt spray resistance. Panels showed good flexibility and no interaction of coating and substrate but salt spray results in 24 hours of exposure showed poor salt spray resistance - the grit blasted panels were in poor condition, the galvanic anodize panels showed no better results than those achieved using tin powder with the same binder.

A similar series of tests was run using the tin-zinc eutectic alloy. Conditions were identical to those listed for the tin-cadmium alloy. Salt spray results were the same with the additional observation that on the galvanic anodized panels more corrosion was observed on those panels that had been exposed to 350°F and 400°F.

⁽³⁾ White, E.L. and F.W. Fink - Corrosion protection of Magnesium and Magnesium alloys DMIC Memorandum 205 Defense Metals Information Center, Battelle Memorial Institute. Columbus, Ohio pg 13

⁽⁴⁾ Greenfield, L.E. and J.S. Bouden <u>Equilibrium Data for Tin Alloys</u>
Tin Research Institute (1949) pg 13 and 53

Ethyl Silicate Coatings

As an additional approach, a series of ethyl silicate based coatings was prepared. Ethyl silicate coatings have advantages due to their low temperature cure, low surface tension, and ability to dissolve partially covalent salts.

Partially hydrolyzed ethyl silicate (20% hydrolyzed) was used in

conjunction with tin powder in the following ratio:

50 ml ethyl silicate (20% hydrolyzed)

50 ml ethanol 200 g tin powder

Using this basic formulation modifications were made based on the similarity to zinc-rich coatings. Additional coatings were made using 1% additions of stannous chloride, and freshly precipitated magnesium fluoride made by reaction of hydrofluoric acid with magnesium nitrate in ethanol. This was also added in 1% (weight/vol.) amount. Another coating was made using the analogy with zinc-rich coatings:

25 ml ethyl silicate (20% hydrolyzed)

25 ml ethanol

4 grams carbon black

6 grams asbestos powder

10 ml 10% solution of stannous chloride in ethanol

120 grams tin powder

This formulation was based on recommendations in reference 5.

One additional ethyl silicate based coating was made, incorporating the tin-cadmium eutectic alloy powder in the ethyl silicate/ethanol based binder using a ratio of 2 grams/milliliter.

All the formulated ethyl silicate based coatings were applied to galvanic anodized panels, allowed to air dry two to three days, then exposed to salt spray for 24 hours. All showed little or no salt spray resistance.

Additions of Salts to Silicate Binders

Attempts were made to incorporate suitable salts into alkaline silicates and quaternary ammonium silicate solutions. Of particular interest was the introduction of soluble fluoride ion which, it was hoped, would react with corroding magnesium alloys producing insoluble magnesium fluoride. Use of sodium fluoride caused coagulation of the silicate binder; however, a solution of 2.5g of potassium fluoride dihydrate was made in SermeTel 421 binder with no coagulation observed. This was used as a binder incorporating 2 grams tin powder per milliliter binder. This was sprayed on galvanic anodized AZ31B panels.

As another possibility, sodium stannate was added to both SermeTel 421 and quaternary ammonium silicate (Binders #1 and #3) producing slow gelation. Additions of 5g of stannic oxide were made to each binder with no noted effect. These were used as binders for tin powder using

⁽⁵⁾ Lopata, Stanley L. and William R. Keithler - Protective Coatings U.S. Patent 3,056,684

the same 2g/ml ratio as before. Resulting coatings were applied to galvanic anodized panels.

As a leaching type constituent strontium chromate was added to SermeTel 421 Binder in the ratio of 20 grams per 100 milliliters. This suspension was ball milled overnight and tin powder added in the 2g/ml ratio. This coating was also sprayed on galvanic anodized panels.

Panels representing all these coatings were dried for 12 hours at 175°F and exposed to salt spray for 24 hours. Panels tested were examined and all found to be in badly corroded condition.

PRIMER COATINGS

Amine Salt Additions

An alternative approach to tin based coatings for magnesium is the use of a primer, preferably a primer that protects due to a leaching constituent. Since chromates, be phosphates, and others are often considered film formers that may retard corrosion and modify the pH of the corroding interface, a series of salts was prepared that incorporated these ions. Since alkali silicate binders are involved it was necessary to use amine salts; alkali silicates are readily gelled or precipitated by most ions. Using amine salts, relatively large amounts of chromate and phosphate ions can be incorporated into alkali silicate binders.

The procedure used was based on 100 ml of SermeTel 421 binder. To this 10ml of 40% methylamine was added followed by 6 grams of chromic acid dissolved in 25 ml of water. Addition of the chromic acid was made slowly with constant stirring. After addition of the acid solution, alkali silicate was added to readjust the silicate concentration.

Using the same procedure binder solutions were prepared incorporating methylamine and phosphoric acid (5% weight/volume) nitric acid (6%) and hydrofluoric acid (1%). In the case of the hydrofluoric acid precipitation eliminated any further additions.

These alkali silicate based coatings incorporating amine salts were mixed with tin powder in the ratio of 2 grams per milliliter and sprayed on galvanic anodized test panels. These were dried at 175°F for 2 hrs. then topcoated with the same coating without the amine salt, i.e. using #1 binder with tin powder.

Panels were subjected to 24 hours of salt spray testing then examined. All were in poor condition with the phosphate containing coating somewhat better but still badly corroded.

Strontium Chromate Primers

A series of panels were prepared utilizing strontium chromate in a primer on galvanic anodized AZ31B alloy:

1. Strontium chromate in SermeTel 421 binder in the ratio of 0.2 grams per milliliter binder.

⁽⁶⁾ ASM Committee on Magnesium, "The Corrosion of Magnesium Alloys Metals" Handbook - Vo. 1, American Society for Metals pg 1088

2. Same as #1 but top coated with a coating consisting of 2 grams tin powder per milliliter of SermeTel 421 Binder.

3. Same as #2 but incorporating 0.1 grams strontium chromate per milliliter of the tin filled topcoat.

These panels were dired at 175°F overnight and exposed to salt spray testing for 24 hours. Results showed all to be leaching heavily and showing coating cracking. Some corrosion of the magnesium alloy was evident.

EDTA Addition

Since salts of ethylenediaminetetraacetic acid (EDTA) are widely used as chelates for alkaline earth ions, a coating was prepared using an addition of 3.1 grams of EDTA tetrasodium salt to 100 milliliters of SermeTel 421 binder. No coagulation was observed. To this binder tin powder was added in the usual 2 grams per milliliter ratio and applied to galvanic anodized test panels then topcoated with a similar coating containing no EDTA salt. These panels were dried at 175°F overnight and exposed to 5% salt spray for 24 hours. Results showed only small pits of corrosion on the test panels, an improvement over previous coatings.

Alkaline Additions

Use of acidic leaching constituents caused little improvement, if any, in the salt spray resistance of tin filled coatings tested. Conversely, use of alkaline leaching materials was tried, to stabilize the pH at a noncorrosive range.

Magnesium oxide, calcium oxide, and aluminum hydroxide were considered possible. Additions were made by mixing the oxide or hydroxide with water and producing a thin paste. This paste, in turn, was incorporated into SermeTel 421 Binder (Binder #1). Immediate precipitation was observed in the case of calcium oxide; the others were compatable with the coating binder.

Using magnesium oxide a slurry was made using 0.15 g of magnesium oxide per milliliter of water; ten milliliters of this slurry was incorporated into 100 milliliters of Binder #1. The silicate content of the binder was readjusted to its correct level and tin powder was added in the usual 2g/ml ratio. This was applied to galvanic anodized panels and allowed to dry at 175°F overnight. The panels were then salt spray tested for 24 hours. Results indicated less observable corrosion than in all previous tests.

Based on this test a series of tests was initiated incorporating magnesium oxide at several concentration levels, aluminum hydroxide additions, strontium chromate and EDTA salts were also included. The following coatings were used:

- 1. 100 ml Binder #1
 1.5 grams magnesium oxide
 200 grams tin powder
- 100 ml Binder #l
 1 grams EDTA tetrasodium salt
 5 grams magnesium oxide
 grams tin powder

- 3. 100 ml Binder #1
 2.0 grams strontium chromate
 1.5 grams magnesium oxide
 200 grams tin powder
- 4. 100 ml Binder #l
 4.5 grams magnesium oxide
 200 grams tin powder

These were applied to AZ31B panels which had been galvanic anodize pretreated and also to bare grit blasted panels. All were cured at 175°F overnight. To determine effectiveness in a limited pore situation these panels were top coated with a clear acrylic finish. Some of the panels were scribed "X", others were unscribed. All were placed in salt spray for 24 hours. Results are listed in Table III.

An additional test was conducted to evaluate the influence of elevated temperature curing on the corrosion resistance of these alkali modified coatings. Coatings #2 and #4 of the preceding group were tested on galvanic anodized panels after curing at 175°F and with two hours at 350°F. Panels were coated with clear acrylic topcoat and then scribed "X" and placed in salt spray for 24 hours. All were badly corroded when examined; the 250°F cured panels were more corroded.

Since potassium permanganate had shown previously a passivating effect on AZ31B panels, coatings were made incorporating Binder #1 saturated with this reagent. Using this binder and tin powder in the 2g/ml ratio two coatings were made; the first incorporating 0.15 g magnesium oxide per milliliter, the second 0.25g of gelatinous aluminum hydroxide. These coatings were applied to galvanic anodized panels and cured at 175°F for 16 hours followed by 24 hours salt spray. Panels showed only light corrosion.

Organic Topcoat Treatment

As a final attempt, a series of 4" x 6" AZ31B-H-O panels were coated with a series of coatings utilizing the alkaline additions after having been given the galvanic anodize pretreatment. These panels were than coated with MIL-P-23377C epoxy-polyamide primer and MIL-C-81773 Polyurethane white topcoat. Coating was done by the Naval Air Development Center, Warminster. Panels were scribed "X" and subjected to cyclic testing - 30 hours 5% salt spray and 16 hours 400°F constituted 1 cycle. Base coatings used were as follows with pretreatments and cures:

CODE	COATING/CURE
BMA	100 milliliters Binder #1
	1.5 grams magnesium oxide
	200 grams tin powder
	applied over grit blasted surface
	cured at 175°F for 2 hours.
B M H	as BMA but cured at 350°F for 2 nours
NONE	as BMA but galvanic anodize pretreatment

TABLE III Salt Spray Results of Tin Filled Alakline Modified Coatings on AZ31B Alloy

Panel #	Pretreatment	Coating	Condition	Results
1 2 3 4	GB GB GA GA	1 + K 1 + K 1 + K 1 + K	0 X 0 X	Badly blistered Badly blistered No corrosion Light corrosion in scribe
5	GA	2 + K	0	No corrosion Badly corroded
6	GA	2 + K	X	
7	GA	3 + K	0	50% of surface corroded 50% of surface corroded
8	GA	3 + K	X	
9	GB	4 + K	0	Badly blistered
10	GB	4 + K	X	Badly blistered
11	GA	4 + K	0	Light corrosion
12	GA	4 + K	X	Corroded in scribe

Code:

Pretreatment: GA - Galvanic Anodize GB - Grit Blast

1-4 as on the previous page
K - Acrylic topcoat Coating:

0 - Unscribed Condition:

X - scribed

CODE (cont.) Coating/Cure (cont.) as above but cured at 350°F for 2 hours GAA 100 ml Binder #1 4.7 grams aluminum hydroxide 200 grams tin powder galvanic anodize pretreatment cured at 175°F for 2 hours. **GAPA** same as GAA only Binder #1 was saturated with potassium permanganate same as BMA only Binder #1 was saturated **GMPA** with potassium permanganate and pretreatment was galvanic anodize 115SC Control panels prepared by NADC, Warminster. Pretreatment was abrasion using alumina inpregnated plastic pads followed by chromate conversion coating per MIL-M-3171C type VIII. 115B Control panels prepared by NADC, Warminster using chromate conversion coating per MIL-M-3171C type VIII applied over grit blasted surfaces

Testing Results

Results are described in Table IV.

The poor test results shown by the coatings tested indicate the following:

- 1. Coatings of the type tested retain moisture even when cured at 350°F causing a blistering effect to be evidenced when top coated with a barrier type coating.
- 2. The leaching effect used modifies the pH of the surface of the coating. This causes a resolution of silicate based coatings, softening them, and spreading corrosion by a "blotter" effect.
- 3. The differences observed between the control panels coated with MIL-M-3171C type VIII conversion coating were considerable. The only variable differing was the extent of surface abrasion prior to the conversion coating. Since the alkaline modified silicate base coats were applied over galvanic anodized panels that had been grit blasted these would show more tendency to corrode due only to their pretreatment. Such silicate coatings do require grit blasting for good adhesion.

CONCLUSION

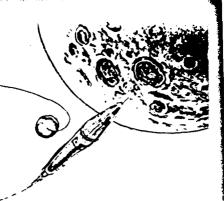
The tests run indicate that use of leaching silicate based coatings containing tin to protect magnesium is not a viable approach. Chromate/Phosphate coatings require a high curing temperature. This cure temperature alone precludes their use. In those tested with tin fillers little corrosion resistance was noted; attack by the coating on the magnesium was evident.

The addition of acidic leaching constituents enhanced corrosion when used with alkali silicate tin coatings. Basic leaching constituents did improve the corrosion resistance of coated panels but not sufficiently to be considered useful.

TABLE IV Results of Heat Cycle/Salt Spray Testing of Topcoated Alkaline Modified Coatings

CODE	RESULTS
ВМА	Large blisters under the coating with severe scribe corrosion
ВМН	Large blisters under the coating, coating pulling away from the scribe
NONE	Large blisters under the coating, severe scribe corrosion
Н	Large coating blisters, servere scribe corrosion
GAA	Few coating blisters, severe coating delamination around scribe
GAPA	Coating is delaminated, servere corrosion
GMPA	Severe blistering, coating is delaminated around scribe
115SC	Slight corrosion in scribe
I15B	Heavy scribe corrosion, corrosion on edges

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July19, 1974

TIN - ZINC

Tin - 92 % .

Zinc - 8 % .

EUTECTIC ALLOYED POWDER , -200 MESH .

Lot # 010 -

Analysis :

Wt./%

Cadmium - 0.002

Iron - 0.005

Lead - - 0.05

Others - 0.14

Hydrogen Loss - 0.14

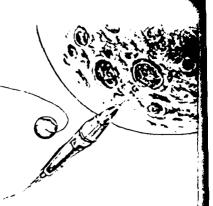
MELTING POINT OF COMPACTED POWDER - 390 DEG. F.

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JULY 29, 1974

TIN, CADMIUM, EUTECTIC ALLOYED POWDER.

Tin - 67 %

Cadmium- 33 % .

Lot # 020, -200 Mesh .

Analysis :

Wt./7.

Lead - 0.07

Copper - 0.03

Iron - 0.1

Zinc - 0.03

Others - 0.12

Hydrogen Loss - 0.1

Melting Point of Compacted Powder - 350 Deg. F

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